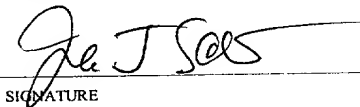


U.S. APPLICATION NO. (if known, see 37 CFR 1.5) 09/582625		INTERNATIONAL APPLICATION NO. PCT/FR99/00319		ATTORNEYS DOCKET NUMBER 00130	
17. <input checked="" type="checkbox"/> The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) : Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$970.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO. \$840.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$760.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$670.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) \$96.00 ENTER APPROPRIATE BASIC FEE AMOUNT =				CALCULATIONS PTO USE ONLY	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	17 - 20 =	0	X \$18.00	\$	
Independent claims	6 - 3 =	3	X \$78.00	\$ 234.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$260.00	\$	
TOTAL OF ABOVE CALCULATIONS =				\$ 1,074.00	
Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28).				\$	
SUBTOTAL =				\$ 1,074.00	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	
TOTAL NATIONAL FEE =				\$ 1,074.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property				\$	
TOTAL FEES ENCLOSED =				\$ 1,074.00	
				Amount to be:	\$
				refunded	\$
				charged	\$
a. <input checked="" type="checkbox"/> A check in the amount of <u>\$ 1,074.00</u> to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>04-0753</u> . A duplicate copy of this sheet is enclosed.					
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.					
SEND ALL CORRESPONDENCE TO Dennison, Scheiner, Schultz & Wakeman 612 Crystal Square 4 1745 Jefferson Davis Highway Arlington, Virginia 22202 (703)412-1155 Ext. 23 (703)412-1161 (Fax)					
				 SIGNATURE Ira J. Schultz NAME 28666 REGISTRATION NUMBER	

FORM PTO-1390
(REV 11-98)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

00130



U.S. APPLICATION NO. (if known) 22338

09/582,025

INTERNATIONAL APPLICATION NO.
PCT/FR99/00319INTERNATIONAL FILING DATE
12 February 1999PRIORITY DATE CLAIMED
13 February 1998TITLE OF INVENTION ALUMINIUM ALLOY STRIPS WITH HIGH SURFACE HOMOGENEITY
AND METHOD FOR MAKING SAMEAPPLICANT(S) FOR DO/EO/US Jean-Luc HOFFMANN; Pierre-Yves MENET; Klaus MAIWALD;
Regine DEBREUX; Martin-Peter SCHMIDT; and Yves SCELLES

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☐ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

09/582625

534 Rec'd PCT/PTC 03 JUL 2000

Dkt. 00130

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

JEAN-LUC HOFFMANN et al

PCT

Serial No.: none assigned
(PCT/FR99/00319)

~~09/582625~~

Filed: Concurrently Herewith

For: ALUMINIUM ALLOY STRIPS WITH HIGH SURFACE
HOMOGENEITY AND METHOD FOR MAKING SAME

PRELIMINARY AMENDMENT AND INFORMATION DISCLOSURE STATEMENT

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Before calculation of the filing fee, please amend
the above-identified application as follows:

IN THE CLAIMS:

Page 24, line 1, change "CLAIMS" to --WHAT IS
CLAIMED IS:--

Claim 7, line 1, change "any of claims 1 to 6" to
--claim 1--.

Claim 8, line 1, change "any of claims 1 to 7" to
--claim 1--.

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Claim 9, line 1, change "any of claims 1 to 7"
to --claim 1--.

Claim 12, line 1, change "any of claims 10 and 11 to
--claim 10--.

Claim 13 (first occurrence), line 1, change "13." to
--21.--.

Claim 13 (second occurrence), line 2, change "any of
claims 1 to 9" to --claim 1--.

Claim 15, line 1, change "any of claims 13 or 14"
to --claim 13--.

Claim 20, line 2, change "any of claims 10 to 16" to
--claim 10--.

Please cancel Claims 16, 17, 18 and 19 without
prejudice or disclaimer of the subject matter thereof.

REMARKS

The claims have been amended to delete all multiple
dependencies, and to delete the use claims.

A duplicate Claim 13 was renumbered Claim 21.

Submitted herewith is the Search Report of the
corresponding PCT application, together with copies of the
references cited therein, which are listed on the attached
Form PTO 1449.

534 Rec'd PCT/PTC 03 JUL 2000

J. Schultz
Ira J. Schultz

[illegible]

703 412-1155

ALUMINIUM ALLOY STRIPS WITH HIGH SURFACE HOMOGENEITY
AND METHOD FOR MAKING SAME

FIELD OF THE INVENTION

The invention relates to aluminium alloy strips with high surface homogeneity, intended for applications requiring a good visual quality or
5 specific optical properties, such as reflectors or anodised plates for construction and decoration. It also relates to a method for making said strips by twin-roll casting.

10 STATE OF THE RELATED ART

Twin-roll casting has been widely used for several decades to manufacture foils or standard sheets made of aluminium alloys. As described in the basic patent FR 1198006 filed in 1958 by Pechiney, it consists of
15 introducing the liquid metal, stored in a supply tank, in the gap between two cooled horizontal rolls rotating in opposite directions, using an injector. The metal is solidified in the form of a continuous strip, while being reduced in thickness due to the roll pressure.
20 These continuous casting machines are very often used

to produce strips between 5 and 12 mm in thickness. In their most recent versions, such as Pechiney Rhenalu's JUMBO 3 CM® casting, they also make it possible to cast thinner strips less than 5 mm thick as described, for example, in the patent FR 2737430.

The strips from these machines are very rarely used as cast. They generally undergo a first cold rolling sequence and, for some applications, a second finishing cold rolling sequence, possibly with special rolls.

These usual twin-roll casting machines make it possible to obtain strips of a homogeneous appearance, but for very demanding applications in terms of surface condition, associated with a surface treatment of the strip liable to reveal existing surface defects or create said defects from metallurgical heterogeneities, e.g. an anodic treatment, chemical or electrolytic brightening, pickling, chemical brightening, cataphoresis or enamelling, the surface quality of strips obtained from twin-roll casting is currently not sufficient. The upper side of the cast strip generally comprises ripples, presented in the form of lines perpendicular to the direction of casting (transverse-longitudinal direction), which appear to be caused by the oscillation of the meniscus of liquid metal during casting. After anodic treatment, these ripples become visible in the form of parallel streaks; this is a visual defect which is conveyed by a difference in the grey levels with a pitch of the order of one to several (e.g. ten) millimetres.

Another surface defect frequently observed consists of parallel mechanical scratches along the strip; this is a roughness defect. The lower side is of usual mill finish quality.

5 In addition to these two types of surface defect, accidental scratching which is not specific to the twin-roll casting technique occasionally occurs.

The need to improve the surface appearance of strips obtained by continuous casting has been
10 expressed for some time and a number of solutions have been proposed.

For example, American patent US 4461152 describes a liquid metal treatment process starting with the injection in the liquid metal of a gas containing
15 chlorine, followed by the passage of the liquid metal through a series of coalescence chambers, and completed with a filtration, thus enabling the reduction of the inclusion rate in the liquid metal, resulting in the improvement of the surface appearance of 5086 and 5182
20 alloy strips for computer disks. However, manufacturers now tend to try to minimise the use of chlorinated gases.

German patent application DE 2443068 of 1974 describes a continuous casting machine between steel
25 belts which aims to improve the surface appearance of aluminium or aluminium alloy strips so as to be able to produce strips for a decorative anodic treatment. The technical solution proposed in said patent application cannot be applied to twin-roll casting, for three
30 reasons:

The surface quality of a strip obtained by continuous casting between belts is intrinsically poorer than with twin-roll casting, which is probably due to belt vibrations. The metal solidification conditions are totally different, since, for continuous casting between belts, the centre of the strip is solidified downstream from the level of the axes of the rolls near the injector, while, for twin-roll casting, it is solidified upstream from this axis. Finally, continuous casting between belts cannot be used to obtain thin strips less than 5 mm thick; consequently, the gap between the rolls is smaller in a twin-roll casting machine.

British patent application GB 2198976 describes an asymmetrical injector device making it possible to increase the casting rate and, therefore, the industrial output of a twin-roll casting machine; the document does not describe an improvement in the surface of the products obtained, which was not the subject of the invention described.

American patent US 5 350 010 aims to optimise the surface quality of strips intended to produce offset printing plates by the precise monitoring of the grain size of the final product, which implies that a certain metal composition is complied with and that certain process parameters, occurring downstream from the continuous casting, such as the cold rolling pass reduction rate, are monitored. Given that the surface defects present on the as cast strip do not generally disappear during the cold rolling operations that follow, this approach, in the applicant's opinion, does

not seem to deal with the cause of the defects but only tries to minimise their consequences on the finished product.

Following a similar technical approach, patent application EP 0821074 also describes a conversion process of a strip obtained by twin-roll casting, enabling the manufacture of offset plates.

The applicant has noted that none of these approaches make it possible to obtain surfaces meeting high optical and visual homogeneity requirements directly by twin-roll casting, possibly followed by several cold rolling passes.

OBJECT OF THE INVENTION

The purpose of the present invention is to obtain, by twin-roll casting, aluminium alloy strips with, on at least one side, high surface homogeneity and that can be used for applications that were previously not open to them. It should be noted that the majority of these applications only absolutely require a very homogeneous surface condition for one of the two sides. Therefore, it is not a problem that the present invention only produces a spectacular improvement in the surface condition for one of the two sides.

The strips according to the invention, cast continuously between two rolls, less than 12 mm thick, preferably less than 5 mm, comprise an upper side, the surface condition of which may be characterised at three different stages of manufacture, corresponding to three types of more or less elaborate industrial products, on specimens subjected to a particular

preparation, representing a typical industrial surface treatment revealing surface defects:

a) The as cast strips show on the surface (excluding accidental mechanical scratching visible to the naked eye) of their upper side, after creating a 1 μm thick anodic oxide layer by sulphur anodic treatment, an optical roughness value S_N , measured on three 5 cm longitudinal sections and three 5 cm transverse sections, such that its mean variation on each section, defined by the ratio:

$$(\text{Maximum } S_N - \text{minimum } S_N) / \text{Mean } S_N$$

is less than 20%, and the difference: $\Delta S_N = S_N \text{ max} - S_N \text{ min}$ is less than 20.

b) The strips after cold rolling to a thickness between 4 and 0.1 mm, preferably between 2 and 0.1 mm, show on the surface of their upper side an optical roughness value S_N , measured under the same conditions, on a specimen having undergone an alkaline pickling treatment on 10 μm , followed by a sulphur anodic treatment resulting in the creation of a 1 μm thick anodic oxide layer, such that its mean variation on each section is less than 20% and ΔS_N is less than 12.

c) The strips that have undergone a first cold rolling followed by a finishing rolling to a roughness $R_a < 0.2 \mu\text{m}$ and brightened electrolytically show on the surface of their upper side, after creating a 1 μm thick anodic oxide layer by sulphur anodic treatment, an optical roughness value S_N such that its mean variation on each section is less than 20% and ΔS_N is less than 3.5 and even 0.5.

The strips according to the invention also have a surface homogeneity such that the 2D roughness range distribution asymmetry value (S_k or skewness parameter), measured using an optical scanner-based technique described below, is between -0.2 and +0.3, and preferentially between -0.1 and +0.2. The 3D roughness (E_k parameter), determined by a mechanical sensor parallel to the direction of rolling according to a method described below, is less than 15, and preferably 8.

The invention also relates to a method for making aluminium alloy strips by continuous casting between two cooled rolls, from a casting tank containing the liquid metal connected to an injector, comprising an upper lip and a lower lip, feeding the liquid metal into the gap between the two rolls, wherein the upper lip of the injector is recessed by at least 2 mm, and preferably by at least 5 mm, with reference to the lower lip.

The level of liquid metal in the casting tank, measured from the median casting level, is kept below 30 mm and preferably below 25 mm.

DESCRIPTION OF THE FIGURES

Figure 1 represents a cross section perpendicular to the roll axis of a twin-roll casting machine, according to the invention.

Figure 2 represents an example of an optical roughness value S_N record along a measurement section for a strip according to the invention of example 1.

Figure 3 represents an example of an optical roughness value S_N record along a measurement section for a strip according to the prior art of example 1.

5 DESCRIPTION OF THE INVENTION

The surface homogeneity of the upper side of the strips is assessed with two different aspects: the presence of roughness defects (mechanical scratching parallel to the longitudinal direction) and the
10 presence of grey level oscillations (streak perpendicular to the longitudinal direction).

To characterise grey level oscillations, three different complementary techniques were used by the applicant:

15 The optical roughness value S_N was measured using a RODENSTOCK RM 400 surface optical measurement system. This device defines and measures S_N between 4 and 100, for surface roughnesses between 5 and 2000 nm. It is based on the principle of the diffusion of radiation by
20 a rough surface. The surface to be evaluated receives an infrared beam, part of which is rediffused, the angular distribution of the diffused rays depending on the morphology of the surface.

The S_N value is measured continuously on 5 cm long
25 sections by sweeping a 0.5 mm diameter beam, and 3 longitudinal sections and 3 transverse sections taken in the same area with a diameter of approximately 10 mm are measured on each specimen. For each section, deviant isolated peaks due to accidental mechanical
30 scratching visible to the naked eye are eliminated, distinguishing them from the ripples to characterise.

From the recorded curve, as shown in figures 2 and 3, the maximum value, minimum value and mean value of S_N , the difference ΔS_N between the minimum value and the maximum value, and the variation of this value, defined by the ratio: $\Delta S_N / S_N$ mean, are determined. The mean of the 6 differences and variations corresponding to the 3 longitudinal and 3 transverse measurements is calculated, giving the mean difference and variation.

Another method to characterise grey level oscillations is by determining the parameter L^*a^*b . This measurement was made using a Minolta ChromaMeter CR-221 colorimeter, with a 3 mm diameter measurement area, a 45° lighting angle and a 0° observation angle. The lighting was supplied by a pulsating xenon bulb. The reference spectrum was IEC spectrum D_{65} . The parameter L^*a^*b was calculated according to the standard ASTM D2244-89, section 6.2. The specimens were characterised by the mean value of the parameter L^* , and by the standard deviation on this parameter. For each specimen, twenty measurements along a generatrix perpendicular to the oscillations were made. This makes it possible with a unilateral risk of 0.05 to differentiate two series of measurements, for which the standard deviation ratio is 2.17. The applicant found that this test makes it possible to reproduce the visual classification of the specimens, except for specimens showing an oscillation pitch significantly lower than the measurement area diameter.

A third method to characterise grey level oscillations is the use of a scanner to obtain the mapping of the grey levels, followed by the two-

dimensional roughness measurement analysis using digital methods known to those skilled in the art.

A metal plate, of a minimum size of 14 cm x 14 cm, and preferably of a size similar to the DIN A4 format, to be characterised is placed on the scanner panel. A perfectly flat calibrated grey level grid used as an internal reference for each measurement is placed beside it. The tests were conducted with a reference grid marketed by Kodak; this grid comprises twenty grey levels progressing in steps of 0.10 included between a white level of density 0.00 and a practically black level of density 1.90. It proved to be necessary to block the white level of density 0.00 so as not to saturate the detection system. A UMAX type scanner was used. This device makes it possible to obtain a resolution of 150 dpi (dots per inch) x 150 dpi with 256 grey levels. It was connected to a PC which recorded the digitised image in grey levels. It proved to be necessary to perform a first calibration scan with a calibration grid alone, to enable the device to select, in automatic mode, the suitable contrast. Then, the digitisation is performed with a specimen and the reference grid. Provided that the calibrated reference grid is always used, those skilled in the art can use other scanner models with at least comparable performances.

Using a software application (in this case, Spyglass Transform 3.02 ©), the area under study was selected interactively; the reference grid and the edges of the remaining image, typically 1 to 3 cm on each edge, and, if necessary, peripheral areas showing

fog or spots not representative of the grey level oscillations to be characterised, were removed. The working area obtained must have a minimum size of 12 cm x 12 cm.

5 The two-dimensional roughness profile is then analysed on seven lines parallel to the direction of rolling (i.e. perpendicular to the grey level oscillation) at least 100 mm long, selected at random in the working area. If the base line of this profile
10 shows a deviation (particularly due to a scanner lighting defect), it is then necessary to rectify it in such a way so as not to affect the roughness itself. This correction may be carried out using the box method, known by specialists, the box size being
15 adjusted so as to reproduce the pitch and range of the profile in an optimal fashion.

The four roughness parameters, known by the specialist, the mean absolute deviation R_a , the mean square deviation R_q , the maximum roughness R_t , and the
20 skewness S_k defined as

$$S_k = \frac{1}{R_q^3} \times \frac{1}{n} \sum_{i=1}^{1=n} (y_i - \bar{y})^3$$

where

$$R_q = \sqrt{\frac{1}{l_m} \int_0^x y^2 dx}$$

and where l_m corresponds to the mean line, given
25 that for an ideal Gaussian distribution, $S_k = 0$, are then calculated.

The applicant observed that for specimens with homogeneous surfaces, the S_k value is between -0.2 and +0.3. To be able, for example, to produce optical

reflectors, an Sk value between -0.1 and +0.2 is preferred. Specimens with non-homogeneous surfaces, corresponding to the prior art, have an Sk value below -0.4. For example, the applicant found values between
 5 -0.45 and -1.38 for specimens of 8011 alloy obtained using the twin-roll casting process according to the prior art.

To characterise roughness defects, a three-dimensional mechanical roughness measurement method was
 10 used. The specimen is placed on a TIXV 200 crossed table. The sensor (Mahr Mesures model FRW 750), with a 5 µm radius of curvature, was conditioned by a Mahr Mesures Perthometer PRK device. The data was recorded using an analogue-digital conversion card in a PC. The
 15 size of the measurement field was 20 mm x 20 mm, with a pitch of 40 µm along x and y. The theoretical depth resolution was given by the maximum range of the sensor (100 µm) and the characteristics of the microprocessor (16 bits), or approximately 0.024 µm. The three-
 20 dimensional roughness was calculated with a software application supplied by Saphir according to the following equations:

$$R_a = \frac{1}{N M} \sum_i^N \sum_j^M |Z_{ij} - Z_0|$$

where N is the number of points along x, M the
 25 number of points along y, Z0 the mean altitude on the observation surface according to

$$Z_0 = \frac{1}{N M} \sum_i^N \sum_j^M Z_{ij} ;$$

$$R_q = \sqrt{\frac{1}{N M} \sum_i^N \sum_j^M |Z_{ij} - Z_0|^2} ;$$

$$S_k = \frac{1}{N M R_q^3} \sum_{i=1}^N \sum_{j=1}^M |Z_{1j} - Z_0|^3 ;$$

$$E_k = \frac{1}{N M R_q^4} \sum_{i=1}^N \sum_{j=1}^M |Z_{1j} - Z_0|^4 ;$$

The parameter E_k , or kurtosis, characterises the flatness of the distribution; it takes the value 3 for an ideal Gaussian distribution.

All the measurements may be made on the upper side, i.e. the side that was in contact with the upper roll, outside areas showing accidental defects such as scratching due to handling or spots, for as cast strips, strips simply cold-rolled or cold-rolled strips then subjected to finishing passes with polished rolls. To be representative of the target application, the measurements are all made on specimens subjected to a sulphur anodic treatment under the following conditions: sulphuric acid concentration 200 g/l, temperature 20°C, voltage 15 V. This treatment results in a 1 µm thick oxide layer. It may be preceded by preliminary alkaline pickling (e.g. at a temperature of 60°C for 7 minutes in a bath containing 50 g/l ALUMINUX 138, a commercially available soda-based product).

For strips produced by twin-roll casting according to the prior art, the mean variation of S_N is greater than 50%, both for as cast strips and for cold-rolled strips. For the strips according to the invention, the mean variation is less than 20% in any case. The difference ΔS_N is less than 20 for untreated casting strips, and less than 12 for cold-rolled strips to a thickness between 4 and 0.1 mm, having undergone alkaline pickling before the anodic treatment. It is

less than 3.5, and even frequently less than 0.5 for strips after the final "polished" cold rolling, i.e. resulting in a roughness R_a less than 0.2 μm , and then brightened electrolytically before the 1 μm anodic treatment layer.

Surprisingly, it was observed that the surface homogeneity of the upper side of twin-roll casting cast strips is improved markedly by a slight modification of the casting machine represented schematically in figure 1.

The machine comprises a casting tank 1 supplied with liquid aluminium alloy and connected to an injector 2, composed of a lower lip 3 and an upper lip 4, feeding the liquid metal in the gap between the two rolls 5 and 6 rotating in opposite directions. The strip 7 comes out of the other side of the gap between the rolls solidified. The modification according to the invention consists of using an injector comprising an upper lip 4 recessed by a distance d with reference to the lower lip 3. This recess d is at least 2 mm and, preferably, at least 5 mm. To prevent this arrangement from inducing an excessive influx of liquid metal in the gap between the two rolls, it is advisable to reduce the metallostatic pressure, i.e. the level of the metal, in the casting tank 1, measured from the median casting level, to less than 30 mm and preferably less than 25 mm, particularly the greater the recess d . The recess of the upper lip of the injector also makes it possible to obtain a more precise positioning of the injector, preventing accidental friction on the roll surface, and thus indirectly improving the surface

condition of the lower side of the cast strip. With a level of metal below 25 mm and an injector upper lip recess of at least 25 mm, the applicant successfully produced polished 1000 and 8000 series alloy products
5 finished by rolling with polished rolls (skin pass rolling) which had identical properties for use as the known products, produced using the more costly semi-continuous casting and hot rolling process. These products could be used to produce flat, folded or drawn
10 light reflectors.

The invention is applicable to all aluminium alloys liable to be cast by twin-roll casting. For example, the applicant obtained good results with some 3000 series alloys and some low-magnesium Al-Mg type
15 alloys such as 5005.

The invention is of particular interest for 1000 and 8000 series AlFeSi alloys, containing 0.01 to 2% by weight of iron and 0.1 to 2% of silicon. Indeed, these alloys, when they are cast by twin-roll casting, show
20 markedly higher mechanical characteristics than those obtained with conventional casting and hot rolling, making their "polished" rolling easier. One of the reasons for the higher mechanical resistance of the strips obtained by continuous casting for this type of
25 alloy is that the quantity of iron in the solid solution in the aluminium is higher. For an alloy containing more than 0.01% (100 ppm) of iron, the quantity of iron in solid solution is greater than 50 ppm + 0.03 x (Fe content in ppm). Another advantage of
30 having a high iron level in solid solution is, for a given iron content, to reduce the intermetallic iron

compounds, the presence of which on the surface is a source of optical defects. For the same reasons, the invention is also of particular interest for low Mg alloys ($Mg < 1.5\%$).

5 In addition, a surface grain size, defined as the mean width of the grains on the surface, measured perpendicular to the direction of rolling by image analysis, less than 20 μm , and frequently 15 μm , is obtained, both on as cast strips and on cold-rolled
10 strips, reducing some appearance defects such as lines. This characteristic of the strips according to the invention is also favourable for subsequent shaping, e.g. by drawing.

15 EXAMPLE 1

An EN AW-1085 alloy (according to the standard NF EN 573-3) was prepared with the following composition (% by weight): Si = 0.040 Fe = 0.038 Cu = 0.0017 Mn = 0.0022 Mg = 0.0032 Zn = 0.002 Ti = 0.02

20 with an addition of 3 kg/t of titanium/boron refining agent. The metal was treated with argon in a Pechiney Rhenalu Alpur ® foundry ladle, and then cast continuously on a Pechiney Rhenalu JUMBO CM ® twin-roll casting machine.

25 The roll diameter was 1150 mm, with a gap between the two rolls of 2.3 mm. The Styrite ® ceramic injector comprised an upper lip recessed by 7 mm with reference to the lower lip, and was supplied by a casting tank with a level of liquid metal of
30 approximately 18 mm. The casting was carried out at a width of 1370 mm, a cast strip thickness of 3.6 mm, a

casting rate of 1.6 m/min and a force between the rolls of 800 t/m of strip width. The strip was then cold-rolled to a thickness of 0.4 mm.

5 Strips with the same composition were also prepared using the standard method consisting of vertical semi-continuous casting, hot rolling of the plates followed by cold rolling to the same thickness of 0.4 mm at two different work hardening rates.

10 The mechanical characteristics of the strips were compared, i.e. the rupture strength R_m (in MPa), the elasticity limit $R_{0.2}$ (in MPa), the stretch (in %) and the work hardening rate n (in %). The results are given in table 1 and show that for this 1085 alloy, according to the invention, values of $R_m > 165$ MPa, $R_{0.2} > 160$ MPa and $A > 6\%$ are obtained.

Table 1

Source	R_m (MPa)	$M_{0.2}$ (MPa)	A (%)	n (%)
JUMBO 3CM continuous casting	173	167	7.7	80
semi-continuous casting	155	148	7	93
semi-continuous casting	165	158	6.2	96

20 It is noted that continuous casting results in both a higher mechanical resistance with a lower work hardening rate, thus making polished rolling easier, and an improved stretch enabling easier shaping.

It was also noted that the surface grain size, determined by image analysis, was 7 μm for strips

according to the invention and 80 μm for strips obtained from vertical semi-continuous casting.

5 Strips with the same composition were also prepared again with Pechiney Rhenalu JUMBO CM $\text{\textcircled{R}}$ twin-roll casting but with an injector according to the prior art with no recess of the upper lip. These strips followed the same process as the strips according to the invention to a thickness of 0.4 mm.

10 The strips according to the invention and the strips obtained by twin-roll casting with an injector according to the prior art then underwent two finishing passes with polished rolls from 0.4 mm to 0.35 mm. After electrolytic brightening followed by anodic treatment with sulphuric acid resulting in a 1 μm thick
15 layer, the optical properties of the strips were measured using the RODENSTOCK RM 400 system. The results are given in table 2.

Table 2

	S_N max	S_N min	S_N max - S_N min	S_N max - S_N min/ S_N mean
Strips according to the invention	7.4	6.4	1	14%
Strips according to the prior art	11	7	4	50%

20 Records of the roughness value on the strips are given in Figs. 2 and 3.

EXAMPLE 2

An EN AW-1070A alloy (according to the standard NF EN 573-3) was prepared: Si = 0.06 Fe = 0.12 Ti = 0.015

with an addition of 1.5 kg/t of titanium/boron refining agent. The metal was cast continuously on the same Pechiney Rhenalu JUMBO 3 CM ® twin-roll casting machine as for example 1.

5 The injector also made of Styrite ® ceramic injector comprised an upper lip recessed by 10 mm with reference to the lower lip, and was supplied by a casting tank with a level of liquid metal of approximately 18 mm. The strip width was 1370 mm, the
10 cast strip thickness 3 mm, the casting rate 2 m/min and a force between the rolls 900 t per metre of strip width.

 The strips cast in this way were then cold-rolled to a thickness of 0.8 mm and underwent two rolling
15 passes with polished rolls to 0.5 mm. Strip specimens were sampled as the process progressed, first 3 mm as cast strips, then 0.8 mm strips after cold rolling and, finally, 0.5 mm after "polished" rolling.

 The 3 mm as cast specimens received a 1 µm thick
20 sulphur anodic treatment. The 0.8 mm strip specimens after cold rolling underwent alkaline pickling on 10 µm followed by a 1 µm thick sulphur anodic treatment. The 0.5 mm strip specimens after polished rolling successively underwent electrolytic brightening and a 1
25 µm thick sulphur anodic treatment.

 Strips with the same composition were also prepared again with Pechiney Rhenalu JUMBO 3 CM ® twin-roll casting but with an injector according to the prior art with no recess of the upper lip. These strips
30 followed the same process as the strips according to the invention up to 0.5 mm and, like said strips,

underwent electrolytic brightening and a 1 μm thick sulphur anodic treatment.

The optical properties of all the specimens were measured using the RODENSTOCK RM 400 system. The results are given in table 3.

Table 3

	Thickness in mm	S_N max	S_N min	S_N max - S_N min	S_N max - S_N min/ S_N mean
Anodised as cast strips, according to the invention	3	53	44	9	19%
Anodised pickled rolled strips, according to the invention	0.8	36	32	4	11%
Anodised "polished" rolled strips, according to the invention	0.5	10	8.5	1.5	17%
Anodised "polished" rolled strips, according to the prior art	0.5	19	13	6	37%

In addition, a surface grain size of 12 μm was measured on the strips according to the invention while the strips with the same composition, subjected to the

same cold rolling procedures but obtained using the conventional process (vertical semi-continuous rolling followed by hot rolling of plates) have surface grain sizes of the order of 70 μm .

5

EXAMPLE 3

For each twin-roll casting, seven different castings were carried out with the following characteristics:

10

Table 4

reference	Alloy	Fe and Si content	casting rate	casting thickness
R1	8011	Fe 0.60; Si 0.75	0.96 m/min	7.0 mm
R3	1050	Fe 0.20; Si 0.14		3.0 mm
R4	1235	Fe 0.37; Si 0.14	2.5 m/min	2.7 mm
R5	1085	see example 1		
R6	8011	Fe 0.70; Si 0.73	1.6 m/min	3.1 mm
R7	1085	see example 1		

The casting R5 corresponds to that in example 1, carried out with the twin-roll casting process according to the invention.

15

The casting R7 corresponds to that in example 1, carried out according to the prior art with a conventional injector.

The other castings were carried out with a twin-roll casting machine similar to that described in example 1.

The results of the optical and roughness measurement characterisations, after pickling and anodic treatment of the untreated casting strips, were as follows:

Table 5
Grey level oscillation characterisation

Reference	Measurement L*a*b		
	L* mean	Standard deviation on L* mean	visual classification (1: best 7: poorest)
R1	74.74	0.26	7
R2	75.47	0.31	3
R3	77.98	0.45	4
R4	79.42	0.20	2
R5	82.50	0.23	1
R6	74.54	0.46	5
R7	74.99	0.77	6

10

Table 6
3D roughness measurement roughness defect characterisation

ref.	Ra μm	Rq μm	Sk	Ek
R1	2.622	4.401	-4.124	35.830
R3	2.111	2.745	-0.784	4.640
R4	3.158	4.060	-0.643	4.364
R5	2.905	3.661	-0.614	3.542

R6	1.759	2.316	-0.856	6.168
R7	2.681	4.033	-2.678	27.002

It is noted that the standard deviation of the parameter L^*mean corresponds to the visual response, except for specimen R1 for which the grey level oscillation pitch is too low with reference to the diameter of the area of an individual measurement. On an industrial level, the specimens R1 and R7 cannot be used for the target applications, since their surface condition is unacceptable in aesthetic and optical terms.

The applicant noted that for the 3D roughness measurement, only the Sk and Ek parameters make it possible to discriminate between the products according to the invention and the products unsuitable for demanding applications in terms of surface appearance.

The applicant also noted that to obtain a surface enabling the target applications, one, or preferentially all, of the following conditions must be met:

- the 3D roughness measurement value of Sk according to the procedure described must be greater than -2.0 and preferentially greater than -1.0;
- the 3D roughness measurement value of Ek must be less than 15 and preferentially less than 8.

The applicant observed that it is preferable, in addition to the one or two conditions mentioned above, for the surface to also have a standard deviation on the value L^*mean less than 0.5 and preferentially less than 0.3.

CLAIMS

1. Aluminium alloy strip with high surface homogeneity produced by twin-roll casting, characterised in that its upper side shows, after a 1 μm thick sulphur anodic treatment, an optical roughness value S_N , measured on three 5 cm longitudinal sections and three 5 cm transverse sections, such that its mean variation on each section, defined by the ratio:

$$(\text{Maximum } S_N - \text{minimum } S_N) / \text{Mean } S_N$$

is less than 20%, and the difference $\Delta S_N = S_N \text{ max} - S_N \text{ min}$ is less than 20.

2. Aluminium alloy strip with high surface homogeneity produced by twin-roll casting and then cold-rolled to a thickness between 4 and 0.1 mm, preferably between 2 and 0.1 mm, characterised in that its upper side, after an acid pickling treatment on a 10 μm thickness, followed by a 1 μm thick sulphur anodic treatment, shows an optical roughness value S_N , measured on three 5 cm longitudinal sections and three 5 cm transverse sections, such that its variation is less than 20% and the difference ΔS_N is less than 12.

3. Aluminium alloy strip with high surface homogeneity produced by twin-roll casting, characterised in that its upper side shows, after pickling and sulphur anodic treatment, at least one of the following characteristics:

(a) an S_k value determined by 3D roughness measurement greater than -2.0 and preferentially greater than -1.0;

(b) an E_k value determined by 3D roughness measurement less than 15 and preferentially less than 8.

4. Strip according to claim 3, characterised in that the standard deviation of the value L^* determined according to ASTM D2244-89, section 6.2, calculated on the basis of 20 individual measurements along a generatrix parallel to the longitudinal direction, is less than 0.5 and preferentially less than 0.3.

5. Aluminium alloy strip with high surface homogeneity produced by twin-roll casting, characterised in that its upper side shows, after pickling and sulphur anodic treatment, an S_k value, obtained by 2D roughness measurement analysis of the images obtained with an optical scanner, between -0.2 and +0.3 and preferentially between -0.1 and +0.2.

6. Aluminium alloy strip with high surface homogeneity produced by twin-roll casting and then cold-rolled to a thickness between 4 and 0.1 mm, preferably between 2 and 0.1 mm having undergone at least one finishing pass with polished cylinders, with a roughness $R_a < 0.2 \mu\text{m}$, characterised in that its upper side, after electrolytic brightening followed by a $1 \mu\text{m}$ thick sulphur anodic treatment, shows an optical roughness value S_N , measured on three 5 cm longitudinal sections and three 5 cm transverse sections, such that its variation is less than 20% and the difference ΔS_N is less than 3.5.

7. Strip according to any of claims 1 to 6, characterised in that it shows, on the surface of its

upper side, a grain size, measured by image analysis, less than 20 μm , preferably less than 15 μm .

8. Strip according to any of claims 1 to 7, characterised in that the aluminium alloy is a 1000 series or 8000 series alloy containing between 0.01 and 2% of iron and between 0.01 and 2% of silicon and that the iron content in solid solution is greater than 50 ppm + 0.03 x ppm total Fe.

9. Strip according to any of claims 1 to 7, characterised in that the aluminium alloy is a 5000 series alloy containing less than 1.5% of Mg.

10. Process for manufacturing a strip with high surface homogeneity by continuous casting between two cooled rolls (5) and (6), from a casting tank (1) containing the liquid metal connected to an injector (2), composed of a lower lip (3) and an upper lip (4), feeding the liquid metal into the gap between the two rolls, characterised in that the upper lip (3) of the injector (2) is recessed by at least 2 mm with reference to the lower lip (4).

11. Process for manufacturing a strip with high surface homogeneity according to claim 10, characterised in that the upper lip (3) of the injector (2) is recessed by at least 5 mm with reference to the lower lip (4).

12. Process according to any of claims 10 and 11, characterised in that the level of liquid metal in the casting tank (1), measured from the median casting level, is less than 30 mm.

13. Process according to claim 12, characterised in that the level of liquid metal in the casting tank

(1), measured from the median casting level, is less than 25 mm.

13. Process for manufacturing a strip according to any of claims 1 to 9 by continuous casting between two cooled rolls (5) and (6), from a casting tank (1) containing the liquid metal connected to an injector (2), comprising a lower lip (3) and an upper lip (4), feeding the liquid metal into the gap between the two rolls, characterised in that the upper lip (3) of the injector (2) is recessed by at least 2 mm with reference to the lower lip (4).

14. Process for manufacturing a strip according to claim 13, characterised in that the upper lip (3) of the injector (2) is recessed by at least 5 mm with reference to the lower lip (4).

15. Process according to any of claims 13 or 14, characterised in that the level of liquid metal in the casting tank (1), measured from the median casting level, is less than 30 mm.

16. Process according to claim 15, characterised in that the level of liquid metal in the casting tank (1), measured from the median casting level, is less than 25 mm.

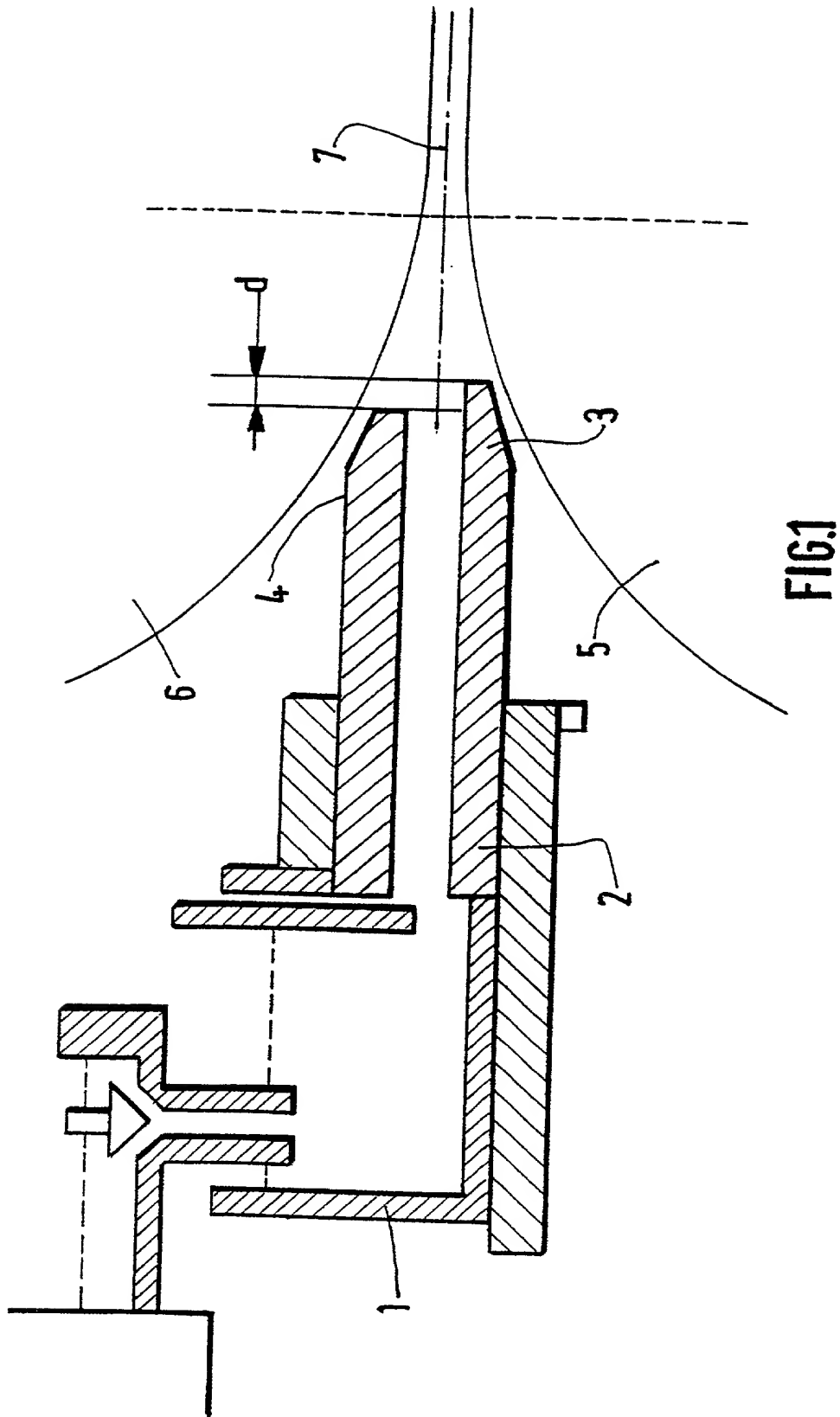
17. Use of a strip according to any of claims 1 to 9 to manufacture optical reflectors.

18. Use of a strip according to any of claims 1 to 9 to manufacture anodised and possible lacquered plates for construction.

19. Use of a strip according to any of claims 1 to 9 to manufacture drawn parts.

20. 1085 alloy strip, produced by twin-roll casting according to any of claims 10 to 16, characterised in that, after one or more cold rolling passes with a total work hardening rate below 85%, it
5 shows at least one of the following groups of properties:

- (a) $R_m > 165 \text{ MPa}$ and $A > 6\%$,
- (b) $R_{0.2} > 160 \text{ MPa}$ and $A > 6\%$



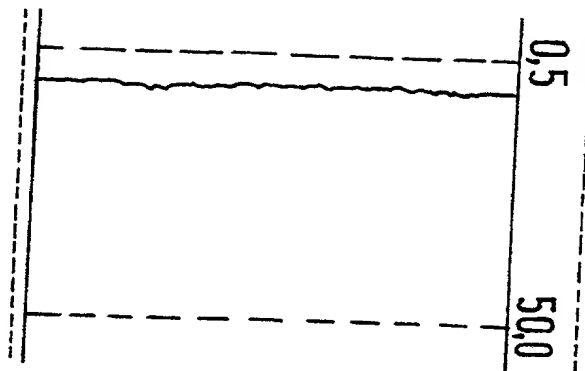


FIG.2

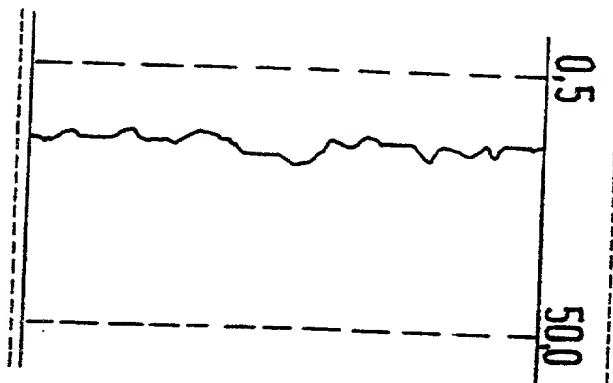


FIG.3

DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION

Docket No. 00130

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled ALUMINIUM ALLOY STRIPS WITH HIGH SURFACE HOMOGENEITY AND METHOD FOR MAKING SAME

specification of which

(check one) XX is described and claimed in PCT International Application PCT/FR99/00319

filed on (MM/DD/YYYY) February 12, 1999 amended on _____

(if applicable)

(OR) _____ is described in United States Application Number _____

filed on (MM/DD/YYYY) _____ (OR) _____ is attached hereto.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56.

I hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, any foreign application for patent or inventor's certificate, or any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Claimed? Yes No
98 01987	France	February 13, 1998	XX
PCT/FR99/00319	PCT	February 12, 1999	XX

I hereby claim the benefit under 35 U.S.C. 119(e) of any United States Provisional Application(s) listed below.

I hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or 365(c) of any PCT International application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. §112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

As a named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

Donald L. Dennison
Burton Scheiner

Reg. No. 19920
Reg. No. 24018

Ira J. Schultz
Scott T. Wakeman

Reg. No. 28666
Reg. No. 37750

DIRECT ALL CORRESPONDENCE TO:
DENNISON, SCHEINER, SCHULTZ & WAKEMAN
1745 Jefferson Davis Highway, Suite 612
Arlington, Virginia 22202-3417

DIRECT TELEPHONE CALLS TO: Ira J. Schultz
(703)412-1155 Ext. 23
(703)412-1161 (fax)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor Jean-Luc HOFFMANN

(First, Middle, Family Name or Surname)

Inventor's signature Jean-Luc HOFFMANN

Date August 1st, 2000

Residence Matzenheim, France

Citizenship France

(City, State, Country)

Full Post Office Address 28, rue JS. Bach, 67150 Matzenheim, France

Full name of second joint inventor Pierre-Yves MENET

(First, Middle, Family Name or Surname)

Second inventor's signature Pierre-Yves MENET

Date August 1st, 2000

Residence Colmar, France

Citizenship France

(City, State, Country)

Full Post Office Address 7, rue Serpentine, 68000 Colmar, France

3-00 Full name of third joint inventor Klaus MAIWALD
(First, Middle, Family Name or Surname)
Third inventor's signature [Signature] Date July 12, 2000
Residence Moirans, France FRX Citizenship German
(City, State, Country)
Full Post Office Address Residence du Parc - Les Chataigniers, 38430 Moirans, France

4-00 Full name of fourth joint inventor Regine DEBREUX
(First, Middle, Family Name or Surname)
Fourth inventor's signature [Signature] Date July 12, 2000
Residence Le Touvet, France FRX Citizenship France
(City, State, Country)
Full Post Office Address 570, Grande Rue, 38660 Le Touvet, France

5-00 Full name of fifth joint inventor Martin-Peter SCHMIDT
(First, Middle, Family Name or Surname)
Fifth inventor's signature [Signature] Date July 12, 2000
Residence La Murette, France FRX Citizenship ~~French~~ German
(City, State, Country)
Full Post Office Address 245, Chemin du Clapier, 38140 La Murette, France

6-00 Full name of sixth joint inventor Yves SCELLES
(First, Middle, Family Name or Surname)
Sixth inventor's signature [Signature] Date July 7, 2000
Residence Vodable, France FRX Citizenship France
(City, State, Country)
Full Post Office Address Le Bourg, 63500 Vodable, France